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Nalco Docket No. 7701  
Customer No. 000049459

In the United States Patent and Trademark Office

Applicants: Brian V. Jenkins et al. ) **Declaration**  
Serial No.: 10/617,467 ) Examiner: Krisanne M. Jastrzab  
Date Filed: July 11, 2003 ) Group Art Unit: 1744

For: METHOD OF INHIBITING CORROSION OF COPPER PLATED OR METALLIZED SURFACES AND CIRCUITRY DURING SEMICONDUCTOR MANUFACTURING PROCESS

DECLARATION OF BRIAN V. JENKINS AND JOHN E. HOOTS  
UNDER 37 C.F.R. § 1.132

Commissioner for Patents  
P. O. Box 1450  
Alexandria, VA 22313-1450

Dear Sir:

Brian V. Jenkins declares that:

1. He is a co-inventor of U.S. Patent Application Serial No. 10/617,467, entitled "METHOD OF INHIBITING CORROSION OF COPPER PLATED OR METALLIZED SURFACES AND CIRCUITRY DURING SEMICONDUCTOR MANUFACTURING PROCESS," which was filed on July 11, 2003 and is now pending.
2. He attended and graduated from Northwestern University in Evanston, Illinois in 1977, receiving a B.S. in Chemical Engineering.
3. He is currently employed as a Global Industry Development Manager for Microelectronics in the Manufacturing Strategic Business Unit at Nalco Company and that his employment with Nalco Company began in October of 1978. Prior to that date he was employed at Turtle Wax, Incorporated in Chicago, Illinois, where he worked in the field of Process Engineering.
4. He has read and understood the above-captioned patent application, the pending Office Action dated April 17, 2006; and

John E. Hoots declares that:

1. He is a co-inventor of U.S. Patent Application Serial No. 10/617,467, entitled "METHOD OF INHIBITING CORROSION OF COPPER PLATED OR METALLIZED SURFACES

Nalco Docket No. 7701  
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AND CIRCUITRY DURING SEMICONDUCTOR MANUFACTURING PROCESS," which was filed on July 11, 2003 and is now pending.

2. He attended and graduated from Millikin University in Decatur, Illinois in May, 1978, receiving a B.A. in Chemistry and Mathematics and that he attended and graduated from the University of Illinois in Champaign-Urbana, Illinois in November, 1983, receiving a Ph.D. in Inorganic Chemistry.
3. He is currently employed as a Research Associate in Sensor Research at Nalco Company and that his employment with Nalco Company began in November of 1983. Prior to that date he was employed at the University of Illinois in Champaign-Urbana, where he worked in the field of Chemistry as Teaching/Research Assistant (1979-1983). Prior to his employment at the University of Illinois he was employed by A.E. Staley Manufacturing Company in Decatur, Illinois, where he worked in the field of Research and Development in Paper Chemistry (1977).
5. He has read and understood the above-captioned patent application, the pending Office Action dated April 17, 2006; and

Both John E. Hoots and Brian V. Jenkins declare that:

1. Accurate measurement of triazole dosage in microelectronics and semi-conductor related applications depend upon a significant number of operating conditions and requirements. These factors present a number of unusual challenges. The excitation and emission wavelengths required for accurate measurements unexpectedly vary in relation to application fluid conditions. Finding acceptable and accurate levels of fluorescence analysis conditions required extensive performance testing. This technology has now been accepted as the method choice in many industry facilities.
2. Challenges of Measuring High Levels of Triazole: Fluorescence measurement is difficult in the presence of high analyte levels and high triazole dosages, such dosages being typically up to 600 ppm in microelectronics applications. High light absorbance levels and fluorescence quenching can also adversely impact accurate fluorescence measurements. Accurate measurement of triazole dosages in these applications required extensive testing of numerous combinations of optical filters to find the correct combination of excitation and emission wavelengths. Further, access to the required range of wavelengths would very likely not have been possible without the recent commercial development of xenon flashlamp-based fluorometers.
3. Challenges of Measuring Low Levels of Triazole: Certain microelectronics applications (e.g., copper-plated silicon wafer rinsing) require triazole dosages of less than 1 ppm to prevent unnecessary triazole discharge. Fluorescence is capable of accurately measuring triazole concentration at dosage levels of 2 to 3 orders of magnitude (i.e., 100 to 1000-fold) lower than light absorbance techniques. Hence, where it was not possible to

Nalco Docket No. 7701  
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accurately measure triazole dosage with light absorbance of previous designs, it is possible with the current fluorescence technique.

4. Challenges of Microelectronics Application Fluid pH: Changes in pH can greatly impact accurate measurement of triazole dosages including the intensity and excitation and emission wavelengths of triazole fluorescence. High purity water, such as in microelectronics applications, are particularly challenging because of the *absence* of pH buffering substances (e.g., phosphate, carbonate, bicarbonate, silicate, and other pH buffers). Such high water purity creates additional challenges that are not present in other triazole applications, such as industrial water systems. Significant pH variation in microelectronics applications creates a unique set of analysis conditions and makes it difficult and nonobvious to accurately measure triazole dosage and to maintain accurate dosage over time.
5. Challenges of Triazole Chemical Composition: Different triazole chemistries (e.g., benzotriazole, tolytriazole, butylbenzotriazole, naphthotriazole, etc.) exhibit distinctive fluorescence, chemical behavior, and response to operating conditions. Accurate fluorescence measurement of different combinations of triazole chemistries, particularly the presence of other operating factors, is nonobvious and required rigorous testing.
6. Challenges of High Purity Fluids: High purity fluids are easily contaminated and specialized flowcell assemblies were developed and tested to ensure fluid compatibility. Over twenty different triazole chemistries were also tested to ensure consistency of fluorescence measurements and flowcell compatibility.
7. Commercial Success, Industry Acceptance, and Long-Felt Need: Conversations with several industry experts revealed a long-felt need for a simple, accurate triazole concentration measurement method. Prior to the present invention, other methods were cumbersome (multiple reagents needed) and substantially challenging and difficult to implement the measurement device into the system. We have heard numerous and consistent comments regarding the ease of installation, integration, operation and superior accuracy of our metrology, as compared to previously available metrologies. Microelectronics facilities that have used other metrologies have conveyed that our fluorescence unit has filled a long-felt but unsolved need.

Dated: 9-15-06Signed: Brian V. Jenkins  
Brian V. JenkinsDated: 9-15-06Signed: John E. Hoots  
John E. Hoots